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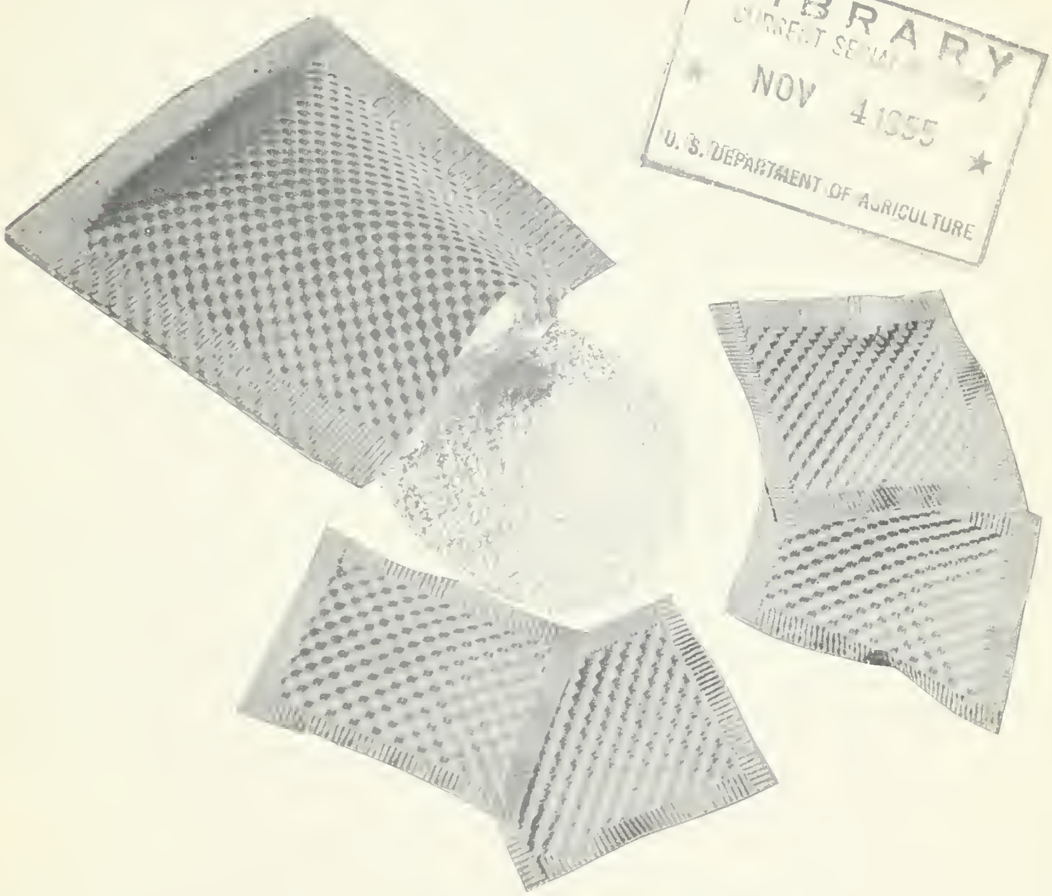
IN-PACKAGE DESICCATION

Studies of Desiccants and Desiccant Containers

Carl E. Hendel, Horace K. Burr and L. Janet Forrester



800 Buchanan St.,
Albany 10, California

Western Utilization Research Branch of Agricultural Research Service

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Contents

IN-PACKAGE DESICCATION--RECENT STUDIES OF DESICCANTS AND DESICCANT CONTAINERS

Carl E. Hendel, Horace K. Burr, and L. Janet Forrester

Abstract. Indented stretchable heat-sealed desiccant bags have resisted failure from expanding calcium oxide desiccant much better than have comparable nonindented bags. Indications are that with such indented bag materials there will not be danger of desiccant-container failure even when unfilled parts of the bag are "pinched-off", at least with desiccants of no more than 90 percent volumetric expansion and with heat seals of 3/8 to 1/2 inch in width.

Twelve samples of calcined limestone were tested for volumetric expansion, activity, and ultimate capacity. They had approximately the same activity and capacity as a sample of recalcined hydrated lime. One of them, in powdered form, had a volumetric expansion of only 70 percent (compared to 60 percent for the recalcined hydrated lime). For the others, the expansion ranged from 110 to 170 percent. It would seem that certain of these calcined limestones would be quite satisfactory as desiccants, particularly if they can be used with indented stretchable desiccant container materials such as those studied in the present work.

In-package desiccation is a process for stabilizing dehydrated foods (or related products) by reducing their moisture content to a very low value after packaging. It consists of enclosing with the product a small packet of a chemical drying agent. There is a subsequent gradual transfer of moisture from the product to the desiccant.

Previous reports from this Branch have described effects of in-package desiccation on the keeping qualities of dry products (3, 4, 6, 7) and the choice of desiccant and desiccant container (1, 2, 10). Recent studies have provided further information that may be helpful to those interested in the use, or in the development, of in-package desiccation. This information is made available because of current interest in tomato and fruit-juice powders recently developed at the Western Utilization Research Branch (5, 8, 9). These powders require in-package desiccation for stability at elevated temperatures.

Desiccant Container

As reported previously (2, 10), calcium oxide desiccants expand with hydration. Under improper conditions they can break the desiccant container. It was stated that with the available desiccant bag materials, care should be taken to avoid "pinching-off" of unfilled parts of the desiccant bag from filled parts. Calcium oxide tends to "bridge" on hydration, and will break the desiccant bag before it will flow into "pinched-off" parts of the bag. This difficulty occurred with stretchable container materials then available as well as nonstretchable materials.

Packaging would be simplified if precautions were not necessary to avoid the pinching-off. Several types of container materials have therefore been tested. They have included paper-coated jean cloth (JC), porulated vinyl sheet (PV), and 3 types of a two-way creped (x-creped) kraft paper.[1] The creping permits the paper to stretch. The three types used were nonindented (N), indented (I), and bleached indented (BI). Indentation provides for expansion more readily under low-tensile stress. These x-creped krafts are heat sealing. The vinyl sheet was electronically sealed. The jean-cloth bags were formed by sewing.

Bags of these materials, containing 140 grams of powdered or pellet-form recalcined hydrated lime, were hydrated according to the rapid procedure of AIC-373 (2) in cut-off No. 10 cans under 3 pounds of sand in partially evacuated desiccators (8 cm. Hg) containing a saturated solution of sodium chloride. The desiccators were not completely evacuated because heating would have resulted from excessively rapid hydration of the desiccant, with resultant weakening of container heat seals. The kraft and porulated vinyl bags were formed by sealing on all 4 sides; those of jean cloth were made by folding the sheet to form one side and by sewing to form the other 3 sides. Unfilled parts of these bags were pinched off by the sand used to simulate the dry product resting on the desiccant container. In all 74 bags were tested under various conditions.

Table 1 shows results of a typical set of comparisons. Before the desiccant bags were placed in the No. 10 cans, they were picked up by one end and the desiccant was shaken into as small a part of the bag as possible without stretching the bag.

On hydration, the jean cloth bags did not break, but the porulated vinyl bags were badly ruptured. Puncture of the porulated vinyl by sharp corners of the desiccant pellets may have initiated the failure of the desiccant containers. Failure did not occur at the seal. The nonindented creped kraft bags ruptured at the heat seals, but the indented krafts did not. Partial separation of the heat seals was less pronounced for the bleached indented than for the indented.

An explanation of the difference in behavior of these materials is suggested by results of mechanical tests (Table 2). These tests were made in a Scott tensile test machine at 70°F. and 65 percent relative humidity.[2] The specimens were strips 1 inch wide by 8 inches in length, cut in both the longitudinal and the transverse direction. The active length (distance between jaws) was 2 inches, and the rate of movement of the lower jaw was 2 inches per minute. Before use,

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1/ The double-creped kraft used was Promset 831X, produced by the Mid-States Gummed Paper Co. of Chicago. Mention of commercial names of materials does not constitute a recommendation by the Department of Agriculture over comparable products not named.

2/ It is recognized that the conditions would have more nearly simulated those of in-package desiccation if a low relative humidity approaching zero could have been used. Unfortunately this was not feasible. However, as indicated in a following paragraph, the results of the mechanical tests do appear helpful in understanding the behavior of heat-sealed stretchable desiccant containers.

Table 1. Resistance to failure of various potential desiccant container materials

JC = paper-coated jean cloth. PV = porulated vinyl. N, I, and BI = nonindented, indented, and bleached indented forms of the x-creped kraft.1/

Desiccant container material	Number of bags tested	Area of bag containing desiccant, inches2/	Bag condition after hydration
JC	2	4x3-3/4	No rupture
PV	3	4x3-1/2 to 3-3/4	3 to 6-inch split
N	2	4-1/4x4	Both bags ruptured due to heat-seal separation3/
I	3	4-1/4x3-1/2	5/16 to 3/8 inch separation of heat seals3/
BI	2	4-1/4x3-1/2	3/16 inch separation of heat seals3/

1/ Desiccant: recalcined hydrated lime pellets of 90 percent volumetric expansion. The unfilled parts of the bags were in approximately the same plane as the parts containing the desiccant.

2/ The overall sizes of the bags were 4x8 inches for the jean cloth, 4.5x6 inches for the porulated vinyl, and 5x7 inches for the kraft.

3/ Width of heat seals was 3/8 inch.

Table 2. Mechanical properties of desiccant bag materials

Material 1/ and direction	Ult. tensile 2/ strength, lb./in. width	Elongation 2/ at rupture, %	Elongation at 2/ 2 lb./in. load, %	Bond 2/ strength, lb./in. width
Nonindented (N)				
Long.	15	40	0.8	1.7
Transv.	9	44	1.0	
Indented (I)				
Long.	13	30	3.8	2.1
Transv.	8	40	8.4	
Bleached (BI) indented				
"A"	5.6	41	35	1.2
"B"	5.2	36	32	
Porulated vinyl (PV)	14	138	16	---

1/ Materials N, I, and BI are all forms of the x-creped kraft. With material BI it was not possible to identify the longitudinal (machine) and transverse directions. The porulated vinyl sheet was essentially isotropic.

2/ Average of 4 to 6 replicate measurements.

the machine was calibrated with dead weights suspended from the upper jaw. Load-deformation curves were automatically recorded. Bond-strength tests were run on x-creped kraft bags that had been fabricated and filled with lime by a custom packaging firm. Strips from the bags gave the following bond strengths when the heat seals were pulled apart (jaw velocity 2 inches per minute): nonindented, 1.7 lb./inch of width; indented, 2.1 lb./inch of width; bleached indented, 1.2 lb./inch of width. Probably no significance should be attached to the variations in bond-strength values.

If the assumption is made that the heat seals and the stretchable paper behave the same way when slowly stretched by the lime in our bag-rupture tests as they did in our tensile and bond-strength tests, it is obvious that the great stretchability of the nonindented material would not be utilized under the conditions of the bag test. That is, the heat seals would begin to fail before the paper had stretched more than 1 to 2 percent. On the other hand, for the indented, and particularly for the bleached indented, the stretch was much more readily available at low load. Bags of these materials should therefore suffer much less separation of the heat seal than bags of the nonindented materials.

Semiquantitative confirmation was obtained by cutting strips from bags in which powdered lime of 70 percent volumetric expansion had been hydrated in rupture tests. Measurements showed that the nonindented material had stretched 1-1/2 to 3 percent, while an additional 5 to 9 percent of width had been made available to the desiccant by partial opening of the heat seals. With indented material, the paper stretched 9 to 13 percent and a slight failure of the heat seals furnished an extra 2 percent of width.

These observations thus show that the stretch is much more readily available with the indented materials. They explain the results of Table 1, where the nonindented bags ruptured (heat-seal separation of more than 3/8 inch), but the bleached indented bags had a seal separation of only 3/16-inch. The conditions for the tests of Table 1 are considered at least as severe as any pinch-off that might occur in normal practice. Hence, it seems that danger of rupture is minimized, even under these conditions, when indented desiccant bag materials of the characteristics of those tested are used. Desiccants that do not expand more than approximately 90 percent on hydration are recommended, with heat seals on the container of about 3/8- to 1/2-inch width (for bags containing 100 to 150 grams of calcium oxide). Increasing the width of the heat-seal would, of course, increase its resistance to rupture.

Since the jean cloth bags did not rupture in the tests reported in Table 1, containers of this type may be usable under pinch-off conditions with desiccants of relatively low volumetric expansion. Data in Table 4 suggest, however, that they could not be so used with calcined limestones of high expansion. Less danger of rupture would exist with suitable stretchable bag materials with the stretch readily available. Economic considerations would also seem to favor heat-seal stretchable materials, since the desiccant bags can be formed and filled on high-speed automatic equipment.

Calcined Limestone and Magnesia

Recalcined hydrated lime has been a preferred desiccant for dehydrated fruit and vegetable products. The preference is based on its relatively low expansion on hydration in comparison with the calcined limestones previously tested. However, its availability in commercial quantities has been uncertain. In an effort to find alternative, cheaper, and possibly better lime desiccants, inquiries were directed to a number of manufacturers of quicklime. They were asked to submit samples which they thought might be particularly active and/or particularly low in expansion during hydration. Twelve samples of lime and one of magnesia (MgO) were received.

Except for samples H and M (Table 3), which came in powder form, each material was broken in a jaw crusher and milled to a coarse powder. A portion of this material was used for the expansion test; the rest was sieved and the fraction passing the 10- and retained on the 20-mesh screens was used for the activity and capacity tests. In general, the methods described in AIC-373 (2) were used.

Activity was determined by exposing duplicate 2-gram samples in a desiccator over a saturated sodium bromide solution and determining the percentage increase in weight after 48 hours. This value is a measure of the rate at which the lime will absorb moisture. The capacity was determined with the same samples by allowing them to hydrate to constant weight. This capacity measures how much moisture the sample will pick up if given sufficient time. In both these tests, the air within the desiccator was gently stirred by a motor-driven propeller, because re-examination of earlier data showed that the measured activity might depend to some extent on the number of samples in the desiccator. It was suspected that the atmosphere of the desiccator might offer enough diffusional resistance to slow down the hydration. With stirring of air any reduction in rate would be avoided.

The volumetric expansion test was carried out with 100-gram samples in 250-ml. graduated beakers. The samples were compacted by repeated tapping of beakers on a table before each volume measurement was made. The results, in Table 3, show that most of the calcined limestones (samples B through L) have activity and capacity that are comparable to those of the recalcined hydrated lime (sample A).

One sample (K) had an expansion very little greater than that of the recalcined hydrated lime (70 vs. 60 percent), which is significant since calcined limestones are more widely available and much less expensive to manufacture than are recalcined limes.

Three of these samples, of widely differing volumetric expansion, were then tested with respect to rupture of desiccant containers. The tests were carried out in the same manner as for the samples of Table 1, with all samples of lime in powder form. Recalcined hydrated lime (sample A) was used as a control.

The data (Table 4) show that for limes with higher volumetric expansions, rupture will occur if the lime is tightly confined, as in the jean cloth bags. With the indented kraft there was an increase in the width of the heat seal opening, from about 1/4 to about 1/2 inch, but there was no rupture. The heat seals were 3/4

Table 3. Activity, capacity, and volumetric expansion of lime and magnesia samples

Sample	Activity 1/	Capacity, 2/ % by wt.	Volumetric expansion 3/ %
A, recalcined hydrated lime	31.9	32.4	60
B, calcined limestone	28.1	30.5	115
C " "	25.2	33.0	150
D " "	31.4	32.9	155
E " "	23.8	29.9	155
F " "	29.2	32.4	170
G " "	32.0	34.2	150
H " "	30.0	31.6	130
I " "	27.3	30.3	110
J " "	27.2	31.2	120
K " "	32.7	33.0	70
L " "	24.8	26.9 4/	115
M, magnesia	7.0	--	-15 5/

1/ Average percent weight gain of duplicate 2-gram samples over saturated NaBr solution in 48 hours.

2/ Approximate ultimate percent weight gain over saturated NaBr.

3/ Percent increase in volume of 100-gram samples hydrated over saturated NaCl solution in evacuated desiccator. Volumes measured after repeated tapping of samples. Averages of duplicate determinations.

4/ Weight gain in 7 days, equilibrium not reached.

5/ After 41 days weight increase was 33.0 percent--equilibrium not reached.

Table 4. Relative tendencies of various calcium oxides to rupture desiccant containers

Calcium oxide 1/	Volumetric expansion %	Indented x-creped kraft (1), greatest width of opening of heat seal, inch 2/	Paper-coated jean cloth (JC)
A	60	4/16, 4/16	No failure
K	70	3/16, 4/16, 4/16	No failure
H	130	4/16, 7/16, 6/16	Both bags failed 3/
F	170	7/16, 8/16, 8/16	Both bags failed 3/

1/ Calcium oxide samples of Table 3. Unfilled portions of desiccant bags were in approximately the same plane as the parts containing the desiccant.

2/ The "width of opening of the heat seal" is obtained by subtracting the final width of the heat seal from the initial width. Thus in this test with heat seals initially of 3/4 inch width, the smallest width of intact heat seal after hydration was 8/16 inch for Sample A, and the width of opening of the heat seal was 4/16 inch.

3/ Failure of the jean-cloth bags was usually in the body of the bag, not at the sewn sides.

Table 5. Expansion of calcium oxide as a function of degree of hydration

	Volume increase 1/				
Weight increase	Recalcined hydrated lime A Pellets	Recalcined hydrated lime A Powder	Calcined limestone F Powder	Calcined limestone H Powder	Calcined limestone K Powder
%	%	%	%	%	%
10	7	11	24	24	13
20	25	32	86	65	41
30	75	54	160	111	78

1/ Desiccant samples (100 grams each) were in 250-ml. graduated beakers, in desiccators evacuated to a pressure of 6 cm. of mercury. The desiccators contained a saturated sodium chloride solution, and the beakers were placed in this solution (not on desiccator plates) to speed dissipation of the heat of hydration (2). Weighing and volume reading, with tapping of beakers to compact the desiccant (2), were at approximately equal 5 percent weight-increase intervals. The volume increases given were read from plots of volume increase vs. weight increase. Each sample was tested in duplicate.

inch in width. The data thus show that rupture is less likely if the desiccant container can readily stretch, as in the indented kraft bags as compared to the jean cloth bags. The width of heat seal opening would no doubt have been less with the bleached indented kraft than with the indented material used because of the greater availability of its stretch at low load (Table 2).

The results thus indicate that use of a stretchable desiccant container material in which the stretch is quite readily available would make possible use of calcined limestones with volumetric expansions greater than could be used in nonstretchable containers. Any new combination of desiccant and desiccant container should, of course, be adequately tested before being used in commercial production.

The magnesia differed sharply from the calcium oxides in that it did not expand on hydration. It was much less active--so much so that the material tested would probably not be a satisfactory in-package desiccant. Further tests are being made with a more active magnesia.

Powdered vs. Pellet Lime

It was found with one lot of recalcined hydrated lime that the volumetric expansion was lower if the lime was reduced to a powder before hydration. For pellets (3/16-inch diameter by approximately 1/4-inch length), the volumetric expansion was 90 percent. For powdered (68 percent passing a 16-mesh sieve, 26 percent passing a 40-mesh), the expansion was 70 percent.

It was found that the difference between 70 and 90 percent expansion can be very important with nonstretchable desiccant containers. A test was carried out comparing the powdered and pelleted samples in jean-cloth bags. The desiccant was shaken into as small a part of the bag as possible without stretching, and the unfilled part of the bag was folded under the filled part. There were 4 bags containing pellets, and 8 containing powder. All of the bags filled with pellets were broken, but there was no failure in any of the bags containing the powdered desiccant. Thus under certain conditions a relatively small increase in volumetric expansion of the desiccant can have serious consequences.

Volumetric Expansion vs. Degree of Hydration

In another test it was found that most of the expansion of recalcined hydrated lime pellets occurs in the latter part of hydration (Table 5). Powdered calcium oxide samples similarly underwent their greatest expansion in the latter part of hydration. Thus danger of container rupture increases markedly as hydration approaches completion. Conversely, the danger of rupture could be much reduced by use of an excess of desiccant.

Summary

Indented stretchable heat-seal bags have resisted failure from expanding calcium oxide desiccant much better than have comparable nonindented bags. Indications are that with such indented bag materials there will not be danger of desiccant container failure even when unfilled parts of the bag are "pinched off", at least with desiccants of no more than 90 percent volumetric expansion.

Failure occurred at the heat seals; resistance to failure can be increased merely by increasing the width of the heat seal.

The use of indented double-creped kraft bags may make it possible to use desiccants with greater volumetric expansion than could safely be used with previously available desiccant containers.

Twelve samples of calcined limestone were tested for volumetric expansion, activity, and ultimate capacity. They had approximately the same activity and capacity as a recalcined hydrated lime. One of them, in powdered form, had a volumetric expansion of only 70 percent (compared to 60 percent for the recalcined hydrated lime). For the others the expansion ranged from 110 to 170 percent. It would seem that certain of these calcined limestones would be quite satisfactory as desiccants, particularly if used with indented stretchable desiccant-container materials such as those studied in the present work. Calcined limestones should be much less expensive than recalcined hydrated limes.

A sample of magnesia was tested. Although low in activity, it had the desirable property of not expanding on hydration.

The expansion of various calcium oxide samples was followed as hydration proceeded. The greatest part of the expansion occurred in the latter stages of the hydration. This result suggests that the danger of container rupture will increase markedly toward the end of desiccation.

Powdering of a recalcined hydrated lime resulted in a decrease in volumetric expansion on hydration. In pellet form the lime had a volumetric expansion of 90 percent; in powder form the expansion was only 70 percent.

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